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XIX.

ON TWO NEW VARIETIES OF VERMICULITES,

WITH A REVISION OF THE OTHER MEMBERS OF THIS GROUP.

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Presented, May 11, 1875.

Since the publication of the writer's first monograph on the vermiculites,* two new varieties of this group of minerals have been brought to his notice by Mr. W. W. Jefferis, of West Chester, Pa., who has most kindly furnished the materials for the following investigation.

The first of these varieties — which occurs at Lerni, Delaware Co., Pa. — has the following characters: The unaltered mineral is of a dull sea-green color, has a highly developed micaceous structure, is an aggregate of rough hexagonal plates, and of very imperfect external form. It is transparent in moderately thin laminæ, and is free from enclosed foreign matter. The optical characters of the mineral closely resemble those of the Culsagee variety of vermiculite, the angle between the optical axes varying in different parts of the same laminæ from 18° to 0°. Its hardness is about 1.5, and three determinations of its specific gravity (taken in alcohol at 23° C.) gave 2.409, 2.368, and 2.373. Heated in a closed tube, it gives off water acid in reaction, changes color, and doubles its volume. Heated before the blowpipe, it fuses to a dirty enamel.

The mineral was prepared for analysis by drying at 100° until its weight was constant, and in this condition was easily decomposed by hydrochloric acid. The bases, after the separation of silica, were converted into nitrates, and separated by Deville's process. The iron and alumina were weighed together as oxides, and the magnesia as the pyrophosphate. The amount and condition of the iron were determined by decomposing the mineral by the process described by the writer in Am. Jour., vol. xliv. page 347, estimating by a standard solution of potassic permanganate the ferrous iron at once, and the total amount of iron after reduction to the ferrous state. Small amounts of lithium and potassium were found by the spectroscope in the residue obtained in Smith's process for the determination of alkalies, but the total amount of alkali probably did not exceed one-tenth of one per cent of the mineral. The mineral, dried at 100°, lost upon ignition,

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* The Vermiculites, their Crystallographic and Chemical Relations to the Micas, by Josiah P. Cooke, Jr. These Proceedings, vol. ix. p. 35. The analytical work in this second paper has been done by Mr. Gooch.

(1.)	(2.)	(3.)	(Mean.)
11.65	11.67	11.71	11.68

per cent of its weight. The details of these determinations will be referred to hereafter. Taking, then, 11.68 as the percentage of water in the mineral, the results of analysis are as follows:—

	(1.)	(2.)	(3.)	(Mean.)	Ratio.
$\ddot{\mathrm{Si}}$	38.17	37.93	38.00	38.03	2.54
$\dddot{\cancel{\mathrm{Al}}}$	12.84	13.07	12.87	12.93	.75
$\dddot{\cancel{\mathrm{Fe}}}$	7.02	7.02	7.02	7.02	.26
$\dot{\mathrm{Fe}}$	.50	.50	.50	.50	.01
$\dot{\mathrm{Mg}}$	——	29.72	29.57	29.64	1.48
$\dot{\cancel{\mathrm{Li}}}$, $\dot{\cancel{\mathrm{K}}}$	——	trace	. . . .	. . . .	
$\dot{\cancel{\mathrm{H}}}$		11.68	11.68	11.68	1.30
		99.92	99.64	99.80	

$$\begin{array}{ccccc}
\ddot{\mathrm{Si}} & : & \overset{\mathrm{VI.}}{\cancel{\mathrm{R}}} + \overset{\mathrm{II.}}{\mathrm{R}} & : & \dot{\cancel{\mathrm{H}}} \\
2.54 & : & 2.50 & : & 1.30 \\
2 & : & 2 & : & 1
\end{array}
\qquad
\begin{array}{ccc}
\overset{\mathrm{VI.}}{\cancel{\mathrm{R}}} & : & \overset{\mathrm{II.}}{\mathrm{R}} \\
1.01 & : & 1.49 \\
2 & : & 3
\end{array}$$

The second of these two varieties of vermiculite occurs at Pelham, Mass. It has a greenish-yellow tint, resembling closely in color the Culsagee variety. In the specimen examined, however, the scales were very much smaller than those of the Culsagee variety, and exhibited no regular outline. Examined with the microscope, they appeared equally free from interpenetrating material. The plates do not show the striation observed both in Culsageeite and Jefferisite. No evidence of macling could be found. The plates were optically biaxial, with a small optical angle and strong negative double refraction like the other vermiculites and magnesian micas. The hardness of the mineral is about 1.5; two determinations of its specific gravity (taken in alcohol at 22° C.) gave 2.160 and 2.161. Heated in a closed tube, it gives off water acid in reaction, changes color, and doubles its volume, and in the forceps before the blowpipe fuses to a dirty enamel.

The mineral, dried at 100°, lost upon ignition

(1.)	(2.)	(3.)	(4.)	(Mean.)
11.21	11.50	11.13	11.43	11.32

per cent of its weight. The details of these determinations will be referred to hereafter.

In (1), (2), (3), and (4) of the following analyses, the ignited mineral was decomposed by fusion with sodic carbonate; in (5) and (6), the mineral, dried at 100°, was decomposed by hydrochloric acid. In all, the ferric and aluminic oxides were weighed together, and the former subsequently determined by reduction and titration with a solution of potassic bichromate. In (4), the iron and alumina were together separated from magnesia by precipitation by ammonia, with the usual precautions. In (5), the bases were converted into nitrates, and alumina and iron separated from magnesia by Deville's process. In both (4) and (5), the magnesia was weighed as the pyrophosphate. A trace only of ferrous iron was found. The spectroscope indicated small amounts of lithium and potassium.*

	(1.)	(2.)	(3.)	(4.)	(5.)	(6.)
$\ddot{\mathrm{Si}}$	41.14	41.28	41.04	40.78	41.27	41.23
$\overset{\cdots}{\overline{\mathrm{Fe}}}$		4.35	4.26	4.49	4.14	
$\overset{\cdots}{\overline{\mathrm{Al}}}$		43.25	43.52	15.05	15.19	
$\dot{\mathrm{Mg}}$				28.82	28.25	
$\dot{\overline{\mathrm{Li}}}$, $\dot{\overline{\mathrm{K}}}$ trace						
$\dot{\overline{\mathrm{H}}}$	11.32	11.32	11.32	11.32	11.32	
		100.20	100.14	100.46	100.17	

Analysis (5), which probably represents the amount of magnesia contained in the mineral more correctly than (4), affords the ratio,

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* In Hallite and Jefferisite, as well as in the Lerni and Pelham varieties, small amounts of lithium were detected. In none of the vermiculites have we been able to detect fluorine.

$$\begin{array}{rrcccc}
 & \ddot{\mathrm{Si}} & : & \dddot{\mathrm{Al}} : \dddot{\mathrm{Fe}} : \dot{\mathrm{Mg}} & : & \dot{\mathrm{H}} \\
 & 2.75 & : & \underbrace{.88 : .17} : 1.41 & : & 1.26 \\
 & 2.75 & : & \underbrace{1.05 : 1.41} & : & 1.26 \\
\text{or} & 2.75 & : & 2.46 & : & 1.26 \\
\text{approximately} & 9 & : & 8 & : & 4
\end{array}$$

The writer has described (loc. cit.) the remarkable hygroscopic properties of the vermiculites, and the difficulty of separating the constitutional from the hygroscopic water which they may contain. The varieties from Lerni and Pelham offer the same difficulty in the determination of their water, thirty to forty hours being required to bring one or two grammes of either of them to a constant weight at 100° C.

In obtaining a constant temperature of 100° C., an electric regulator was used which differs from other similar forms of apparatus in simplicity of construction. The current is made or broken by a very slight rise or fall of mercury in a U tube connected with a glass bulb within the air-bath. By means of a pressure tap which closes an open L of the connecting tube, the air within the bulb may be confined as soon as the bath reaches the required temperature. After this a very slight increase of temperature raises the mercury column sufficiently to close the electric circuit, and then the current shuts the cock which regulates the supply of gas to the burner under the bath. The chief advantage and the novelty of the apparatus is to be found in the simplicity of this stop-cock, which was suggested by Professor H. B. Hill. It consists of an ordinary chloride of calcium tube placed horizontally, and closed at the larger end by a rubber stopper which allows a considerable freedom of motion to a smaller glass tube passing through it; by this the illuminating gas enters the chloride of calcium tube, from which it passes to the burner. When the current is closed, an electro-magnet acting on an armature attached to the outer end of the small tube plunges the curved inner end beneath the surface of some mercury in the bulb of the chloride of calcium tube, and thus shuts off the main supply of gas; although a small orifice in the side of the inner tube allows a sufficient flow to keep the flame under the air-bath alive. The variation of the temperature of the air-bath does not ordinarily exceed one or two degrees during periods of fifteen to twenty hours, even under great variations of pressure in the gas mains.

Table I. shows the percentages of water found in air-dried specimens of the Lerni vermiculite, and Table II. shows the percentages of water found in the same specimens dried at 100° C.

I.

	(1.)	(2.)
Loss at 100° C.	5.75	8.12
„ about 300° C.	10.97	
„ red heat . . .	. . . . . .	8.60
	16.72	16.72

II.

	(1.)	(2.)	(3.)	(Mean.)
Loss at about 300°. .	11.65	2.54	11.71	
„ red heat . . .		9.13		
	11.65	11.67	11.71	11.68

Table III. gives the percentages of water found in air-dried specimens of the Pelham vermiculite, and Table IV. gives the percentages of water found in the same specimens dried at 100° C.

III.

	(1.)	(2.)	(3.)	(4.)
Loss at 100° C.	10.83	10.72		10.80
„ about 300° C.	4.35	10.27		10.14
„ red heat	5.65			
	20.83	20.99		20.94

IV.

	(1.)	(2.)	(3)	(4.)	(Mean.)
Loss at about 300° . . .	4.90	11.50	11.13	11.43	
„ red heat . . .	6.31				
	11.21	11.50	11.13	11.43	11.32

The marked hygroscopic character of the Culsagee, Lerni, and Pelham vermiculites led to the idea that the discrepancies between the published analyses of Jefferisite and Hallite might be due to the hygroscopic nature of these minerals. The water contained in each of them was therefore carefully again determined. Table V. gives the percentages of water found in air-dried specimens of Jefferisite ; analyses (1), (2) and (4) of Table VI. give the percentages of water found in the same specimens dried at 100° ; (3 *a*) gives the percentages of water found in the mineral dried for three months over sulphuric acid, and (3 *b*) the percentages found in the same specimen subsequently dried at 100° C.

V.

	(1.)	(2.)	(3.)	(4.)
Loss at 100° C.	10.28	9.66		10.17
„ about 300 C.	9.58	4.24		9.36
„ red heat		5.43		
	19.86	19.33		19.53

VI.

	(1.)	(2.)	(3 *a*.)	(3 *b*.)	(4.)
Loss at about 300° C.	10.47	4.70	4.46	4.31	10.42
„ red heat		6.01	6.20	6.14	
	10.47	10.71	10.66	10.45	10.42

The mean of (1), (2), (3 *b*), and (4) of Table VI. is 10.51.

It would appear from analysis (3) that the amounts of water lost at 100° C. and over sulphuric acid, during a period of three months, are very nearly identical. Assuming that 10.51 (the mean of the four determinations above) represents the percentage of water in the mineral, dried at 100°, the following scheme contains the published analyses of Jefferisite reduced to this basis. Analyses (1), (2), and (3) are those of Prof. Brush, Dr. Koenig, and Mr. Thomas M. Chatard respectively: —

	Si	$\ddot{\bar{\text{A}}}\text{l}$	$\dddot{\bar{\text{F}}}\text{e}$	$\dot{\bar{\text{F}}}\text{e}$	$\dot{\text{M}}\text{g}$	$\dot{\text{C}}\text{a}$	$\dot{\bar{\text{K}}}$	$\dot{\bar{\text{H}}}$	Total.
(1.)	38.47	18.24	10.92	1.31	20.38	.56	.45	10.51	100.89

$$\text{Ratio}\begin{cases} 2.56 \quad \underbrace{\underbrace{1.06 \quad .34}_{1.40} \quad \underbrace{.04 \quad 1.02 \quad .02 \quad .01}_{1.09}} \quad 1.17 \\ 2.56 \quad : \quad 2.49 \quad : \quad 1.17 \end{cases}$$

(2.)	37.25	19.87	8.17	2.36	21.51	—	—	10.51	99.67

$$\text{Ratio}\begin{cases} 2.48 \quad \underbrace{\underbrace{1.16 \quad .31}_{1.47} \quad \underbrace{.06 \quad 1.07}_{1.13}} \quad 1.17 \\ 2.48 \quad : \quad 2.60 \quad : \quad 1.17 \end{cases}$$

(3.)	38.04	18.39	8.84	2.33	21.34	—	—	10.51	99.45

$$\text{Ratio}\left\{\begin{array}{l} 2.56 \quad \underbrace{\underbrace{1.07 \quad .33}_{1.40} \quad \underbrace{.06 \quad 1.07}_{1.13}}_{} \qquad 1.17 \\ 2.56 \quad : \quad 2.53 \quad : \quad 1.17 \end{array}\right.$$

Table VII. gives the percentages of water found in air-dried specimens of the green variety of Hallite, and Table VIII. gives the percentages of water found in the same specimens dried at 100°. In analysis (4) the mineral was dried for three months over sulphuric acid, and when subsequently heated to 100° for twelve hours met with no appreciable loss.

VII.

	(1.)	(2.)	(3.)	(4.)	(Mean.)
Loss at 100°	3.48	3.19	2.86	2.85	
„ about 300°	12.38	12.86	2.03	2.28	
„ red heat			10.77	10.55	
	15.86	16.05	15.66	15.68	15.81

VIII.

	(1.)	(2.)	(3.)	(4.)
Loss at about 300°	12.82	13.29	2.09	2.44
„ red heat			11.09	10.77
	12.83	13.29	13.18	13.21

The mean of (2), (3), and (4), which agree closely, is 13.23. Reducing therefore Mr. C. E. Munroe's analysis of this same variety to this basis, the following scheme represents the constitution of the mineral dried at 100°.

$\ddot{\text{Si}}$	$\dddot{\bar{\text{Al}}}$	$\dddot{\bar{\text{Fe}}}$	$\dot{\text{Fe}}$	$\dot{\text{Mg}}$	$\dot{\text{K}}$	$\dot{\text{H}}$	
36.34	7.54	8.89	1.14	31.84	.47	13.23	99.45

$$\text{Ratio}\left\{\begin{array}{l} 2.42 \quad \underbrace{\underbrace{.44 \quad .33}_{.77} \quad \underbrace{.03 \quad 1.59 \quad .01}_{1.63}}_{} \qquad 1.47 \\ 2.42 \quad : \quad 2.40 \quad : \quad 1.47 \end{array}\right.$$

It will be noticed, however, that in the case of Hallite there appears to be a constant condition of hydration at about 300°, and that in two experiments the air-dried mineral lost above this temperature 10.77 and

10.55 per cent of its weight. The mean of these values is 10.66; and regarding this as the water of crystallization of the mineral, and reducing Mr. Munroe's analyses accordingly, we obtain the following results : —

$\ddot{Si}$	$\dddot{\bar{Al}}$	$\dddot{\bar{Fe}}$	$\dot{Fe}$	$\dot{Mg}$	$\dot{\bar{K}}$	$\dot{\bar{H}}$
37.17	7.72	9.06	1.18	32.57	.48	11.24
2.48	.45	.34	.03	1.62	.01	1.25
	.79		1.66			
	2.45					
2.48	:	2.45		:		1.25

It would appear, then, that Hallite at 300° is in the same condition of hydration which the other vermiculites examined assume at or about 100°. Now, corresponding to this, there is a very marked fact, indicated by the tables given above, which is worthy of special notice. Air-dried Jefferisite loses, at 100°, about ten per cent of its weight, while air-dried Hallite loses only about three per cent, showing that it holds its water much more firmly than the first. In order to institute a just comparison between the different vermiculites, it is obviously important to seek for each variety the point at which the mineral assumes a constant condition, and maintains it through a considerable variation of temperature. Save only some practical convenience, there is no peculiar virtue in 100°, as the temperature at which a mineral should be dried for analysis. As in the case of crystalline salts we should expect to find for each hydrous mineral a certain point or points of temperature at which it loses the whole, or a part, of its water of crystallization, and certain limits between which it maintains a constant composition. Moreover we should expect that these temperatures would be the more definite in proportion as what we may call the hygroscopic power is the more marked; that, while in some cases the mineral would lose its water at a nearly constant temperature, and the intervals of definite hydration would be well marked, in others the loss would extend over a considerable range of temperature, and it would be more difficult to secure the states of definite composition. That such differences as these are conspicuous among the vermiculites the tables given above abundantly show ; but, in addition to this evidence, the difference in the behavior of the several varieties, when heated, impressed upon us more strongly the principle we have stated than the figures would indicate. Nevertheless, as the following table shows, we have been able to bring all the vermiculites to essentially the same condition. The table is merely a summing up of the results already given, and exhibits a comparison of the atomic ratios * of the several varieties.

	IV. Si.		VI. II. Ɍ + R.		II. Ħ.	VI. Ɍ.		II. R.
Hallite, at about 300° C.	2.49	:	2.47	:	1.25	.77		1.63
	2	:	2	:	1	1	:	2
Pelham Vermiculite, at 100°	2.75	:	2.46	:	1.23	1.05	:	1.43
	2	:	2	:	1	2	:	3
Lerni „ „ „	2.54	:	2.50	:	1.30	1.01	:	1.49
	2	:	2	:	1	2	:	3
Culsagee „ „ „	2.50	:	2.66	:	1.23	1.37	:	1.27
	2	:	2	:	1	1	:	1
Millbury „ (Crossley)	2.38	:	2.74	:	1.14	1.37	:	1.37
	2	:	2	:	1	1	:	1
Jefferisite „ at 100°	2.56	:	2.53	:	1.17	1.40	:	1.13
	2	:	2	:	1	3	:	2

In the next table we bring together some further noteworthy results, of which the details have already been given, indicating that in the case of three, at least, of the vermiculites we have evidence of different degrees of hydration corresponding to different temperatures: —

	IV. Si.		VI. II. Ɍ + R.		II Ħ.						
Atomic Ratio of Hallite Air-dried	2.35	:	2.34	:	1.76	or	8	:	8	:	6
„ „ „ at 100°	2.42	:	2.41	:	1.47	or	8	:	8	:	5
„ „ „ at 300°	2.48	:	2.45	:	1.25	or	8	:	8	:	4
Atomic Ratio of Pelhamite Air-dried	2.45	:	2.19	:	2.32	or	4	:	4	:	4
„ „ „ at 100°	2.75	:	2.46	:	1.26	or	4	:	4	:	2
„ „ „ at 300°	2.90	:	2.59	:	.74	or	4	:	4	:	1
Atomic Ratio of Jefferisite Air-dried	2.30	:	2.28	:	2.17	or	4	:	4	:	4
„ „ „ at 100°	2.56	:	2.53	:	1.17	or	4	:	4	:	2
„ „ „ at 300°	2.68	:	2.65	:	.70	or	4	:	4	:	1

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* The atomic ratio is the same ratio which in most works on Mineralogy is still called the oxygen ratio. The numbers given in this paper are found by dividing the per cent of each oxide by a divisor, which is the quotient of the molecular weight of the oxide divided by the quantivalence of the radical. See writer's Chemical Philosophy, page 450, or Paper on Atomic Ratios, Am. Jour., vol. xlvii., May, 1869.

In the last two varieties the ratios correspond to the symbols: —

$$\begin{array}{llll} \overset{\text{VIII.}}{\mathrm{Si}_2} & \overset{\text{VIII.}}{\mathrm{O}_8} & \overset{\text{VIII.}}{(\text{Ɍ}, \mathrm{R})} & .\ 4\mathrm{H}_2\mathrm{O} \\ & \text{do.} & & .\ 2\mathrm{H}_2\mathrm{O} \\ & \text{do.} & & .\ \mathrm{H}_2\mathrm{O}. \end{array}$$

Here again it will be noticed that the differences in the definiteness of these ratios correspond to the differences of hygroscopic power described above. In the case of Hallite, the ratios are almost precisely those of even molecules, while in the case of Jefferisite the agreement is much less close.

In conclusion, we consider that the following general results may be deduced from this investigation: —

First. That all the vermiculites are unisilicates.

Secondly. That these minerals combine with water in several definite proportions, thus confirming the opinion advanced by the writer in his previous paper on this subject, that the water in the vermiculites is water of crystallization.

Thirdly. That all these minerals may be reduced to the condition expressed by the ratio 2 : 2 : 1, which we regard as the normal ratio of the vermiculites.

Fourthly. That the only essential difference between the different varieties of vermiculites is in the ratio between the sesquioxide and protoxide bases.